RECENT DEVELOPMENTS IN THE RECOVERY OF VOLATILE FRUIT CONCENTRATES

By Roderick K. Eskew

Eastern Regional Research Laboratory[1]

Philadelphia 18, Pennsylvania

It was about 3000 years ago that King Solomon is reported to have said, "Stay me with flagons and comfort me with apples for I am weary of love." I don't know what kind of apples Solomon referred to, but I think you will agree that it is a very gratifying experience to bite into a cold, crisp McIntosh apple and inhale its aroma or to breathe the fragrance of sun-warmed Concord grapes. It is this fragrance about which I wish to speak for it is the attribute without which no fruit product can possess the full-flavor of the parent fruit. Many of you know something of the work that has been done at the Eastern Regional Research Laboratory of the Department of Agriculture in the field of volatile fruit concentrates. I would like to give you a brief history of the development of the basic process and to bring you up-to-date on some of the more recent improvements that have extended its utility. I like the term "volatile fruit concentrate;" it is self-descriptive. But when you have repeated it half a dozen times you have "said a mouth full." So with your indulgence I'm going to borrow a word from your industry and use it in an unfamiliar sense. That word is "essence" and for the purposes of this talk I will use it synonymously with volatile fruit concentrate, ie., a distilled aqueous solution of aromas more concentrated than in the parent fruit.

For years men have tried to capture and hold the elusive fragrance of fresh fruits—usually with disappointing results. Our work in this field was undertaken with the object of obtaining the aromas in concentrated, unaltered form. This would enable their restoration to juice concentrate which would yield the equivalent of fresh fruit juice upon the mere addition of water. Early investigators concentrated the fresh fruit juice under vacuum and attempted to recover the volatile flavors by condensing the vapors at low temperature; perhaps redistilling the condensate to further concentrate flavor. Although logical from the viewpoint of avoiding flavor damage, the use of vacuum has one important drawback. All fruit juices contain dissolved air or other gases and in any flavor recovery system they must be vented since they do not condense. Unless special precautions are taken, such as I will discuss later, this gas leaves the system carrying some aroma with it. The loss of volatiles in the vent gas is approximately in inverse proportion to the absolute pressure at which the system is operated. For example, when the gases are vented from a system under a 27" vacuum (3" absolute pressure) the losses are more than ten times as great as when venting at atmospheric pressure. This is true even when the gases are in both cases chilled and inleakage of air is neglected. In practice there would be some inleakage of air which would further exaggerate this difference. This factor of vent gas loss can be serious when the amount of vent gas is high as a consequence, for example, of a slight fermentation of the juice. Vent gas losses are also magnified with increasing concentration of the distillate and of course with increase in volatility of the product.

[1]One of the Laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

It is always the top notes which are first lost in such a system, throwing the flavor out of balance.

Early Design

With these objections to vacuum in mind, Millerville and coworkers at the Eastern Laboratory in 1944 designed a unit to operate at atmospheric pressure and above.[2] I think I can safely say that with this apparatus they were the first to recover apple essence in concentrated substantially unaltered form. This enabled us to make a full-flavor apple juice concentrate which gave a delightfully fresh flavor on reconstitution. The following slide shows the arrangement of this apparatus. I want to devote a few minutes to it because it was the progenitor of many flavor recovery units of improved design. The juice, in this case apple juice, was fed at a constant rate to a super-heater wherein its temperature was raised in about 3 seconds to 320°F. Flashing the heated juice to atmospheric pressure was sufficient to vaporize about 10%. Previous investigators had shown that all the volatiles in apple juice appeared in the first 10% evaporated. The mixture of liquid and vapor were separated in a vapor-liquid separator, the stripped juice passing to a vacuum evaporator for rapid cooling and concentration. The vapors now concentrated about 10-fold, passed to a fractionating column for further concentration to the desired degree. Usually this was 100-fold, that is, the aroma was collected in a volume corresponding to 1/100 that of the starting juice. The vapors

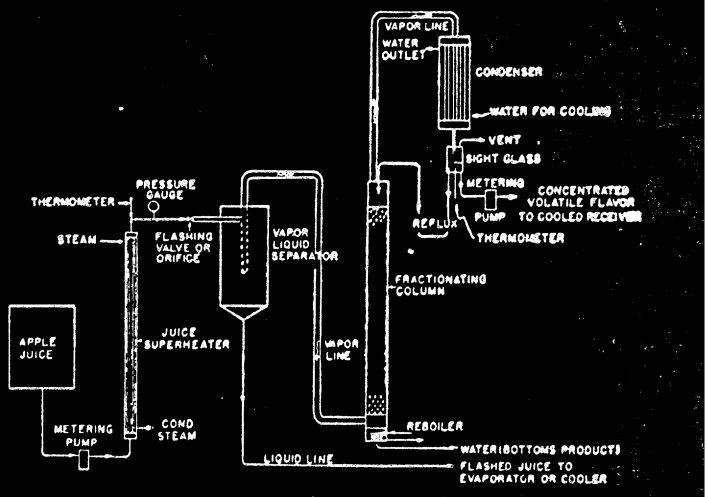

Figure 1. Apple Essence Recovery As Developed at the Eastern Regional Research Laboratory About 1944.

issuing from the top of the column were condensed, a part being returned as reflux and the remainder being drawn off at a metered rate with respect to the juice feed rate. The product was apple essence which when restored to the concentrated stripped juice gave the full-flavor product mentioned above.

Our success with apples suggested that volatile flavor recovery might have much broader applications. However,

[2] "Recovery and Utilization of Natural Apple Flavors", by Howard Milleville and Roderick K. Askew, AIC-63, September 1944. Also in the Fruit Products Journal and American Food Manufacturer, vol. 26, p. 48-51, October 1944.

although the basic principles of the process were found to be applicable to the juice of many other fruits, the early apparatus designs gave mediocre results in most cases. Even when a rapid evaporator was substituted for a superheater and when the vent gases leaving the system were thoroughly chilled with the volatile fruit concentrate before being vented, the apparatus was still unsatisfactory for some fruit juices. In some cases the juice flavor was altered by the heat incident to vaporizing the volatiles and in others the desirable top notes were lost.[3]

Improved Design Extends Utility

The next slide shows an arrangement which is more versatile than the earlier designs and which can be used with all but the most heat-sensitive juices. The juice is fed by a positive delivery pump at a constant rate to an atmospheric preheater where its temperature is brought approximately to its boiling point. It then passes into the vaporizer where the amount required for aroma release is vaporized. This varies widely depending upon the fruit. For apples it is 8 or 10% and for grape juice, 30 or more percent. This separation of

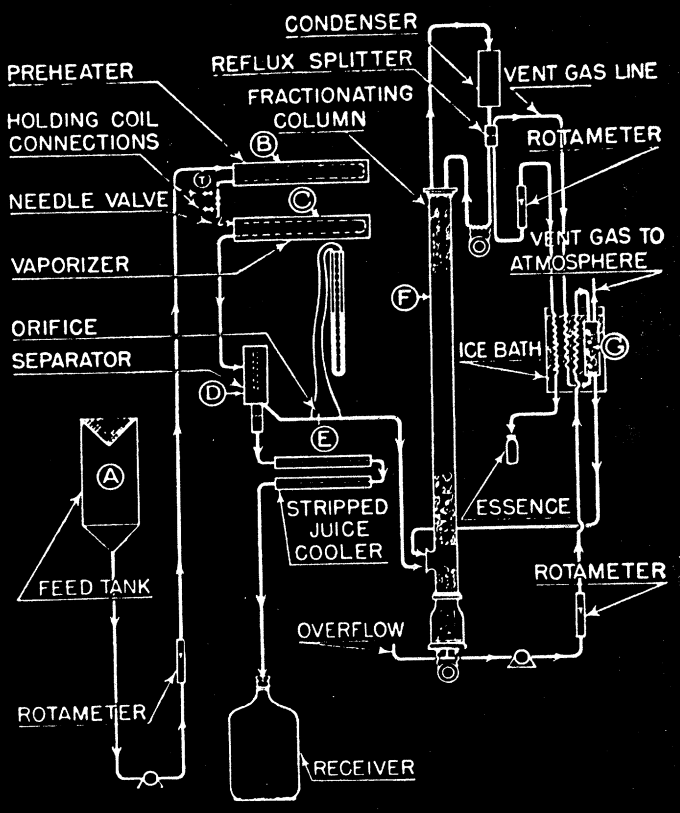

Figure 2. Experimental Unit of Improved Design for More Effective Essence Recovery.

the heating and vaporization steps permits accomplishing the aroma release with the minimum heat effect. The preheater consists of a small diameter tube through which the juice travels at such velocity that it is in turbulent flow and in

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[3] "New Progress in Fruit Flavor Recovery," by Edward L. Griffin, Jr., Lyle L. Davis, Nelson H. Eisenhardt and Margaret E. Heller, Food Industries, Vol. 21, pp. 1545-1547; 1694, 1696, November 1949.

consequence is rapidly and uniformly heated. If the velocity in this tube is not less than 20 feet a second, fouling of the tube walls which resulted with apple juice in earlier designs, is eliminated. In this arrangement the juice can be heated, the desired percentage vaporized, and the juice again cooled by flashing into vacuum in a total time of about 3 seconds. This is very much faster and hence there is less heat damage than in earlier designs.

The aroma-bearing vapors enter a fractionating column where they are concentrated. They are then condensed and a portion is drawn off as product. You will note, however, that there is quite a different method of handling the non-condensable gases before they are vented. Instead of releasing them directly to the atmosphere or chilling them with essence as was done in earlier designs, they are countercurrently scrubbed with cold water. In this case the scrubbing liquid is column bottoms. This means they leave the system in equilibrium with water and hence are free of aroma. In earlier designs when the gases were cooled with essence they were vented in equilibrium with aroma-rich liquid. The scrubbing liquid, containing whatever top notes it has picked up from the vent gases, is returned to the column. Because of this liquid feed, we require a stripping section in the column where it was not formerly necessary. We have used this arrangement with good success in making essences from apple, grape and cherries. It should be applicable to many other juices and will permit the making of highfold essences with good recovery because of the system used in treating the vent gas.

Vacuum Operation Required for Some Juices

There are, however, juices which are too heat sensitive to be handled in this way, notably orange and strawberry. The whole system must then be operated under vacuum. This isn't merely a matter of attaching a source of vacuum to a unit such as I have just described. Although the basic steps in the process of flavor recovery remain the same, the apparatus must be modified chiefly as regards the relative size of its component parts. For instance, if the system is to operate at the same juice rate and under 3 inches absolute pressure, the fractionating column and some other vapor handling parts must be enlarged to accommodate about 10 times the volume of vapor that they were required to handle at atmospheric pressure.

With vacuum operation we must, of course, be especially careful to avoid aroma loss in the vent gas. This can be prevented by scrubbing the gas with chilled column bottoms as shown in the last slide, although of course the scrubbing system must be enlarged to accommodate the increased volume of gas at the lower pressure. We built such a unit on a pilot-plant scale. It was used at our Bureau's Winter Haven, Florida, laboratory and could strip and concentrate the aroma of orange juice without heat damage to the juice.[4] However, there is some question as to the practical value of the essence so recovered. We have recently constructed another vacuum unit and will soon use it on strawberry juice.

Heat Effect Sometimes Desirable

In contrast to juices which must be handled under vacuum, there are others in which the desired flavor can be intensified by deliberate heating. Montmorency and Morello cherry juices are in this category. We have found that if Montmorency cherry juice is heated for 1¼ minutes at 230°F. it will develop more aroma. If the aroma is then stripped and restored to the juice concentrate the product will have much more cherry flavor than if made from unheated juice. This isn't very surprising for we all know good cherry pie has more of what we call cherry flavor than does the raw fruit itself.

Concord grape juice also benefits flavor-wise from some degree of heat. Flavor recovery studies made at various stages of conventional manufacture show a progressive flavor enhancement up to and through the aging process for tartrate settling. Undoubtedly some other fruits would be better for heat treatment. We are all familiar with flavor development by the roasting of coffee beans, the fermentation of vanilla pods, the aging of whisky and the cooking of many foods. I wonder if we have yet exhausted the possibilities of flavor enhancement (and I mean either modification or intensification) in the case of fruits. I am told that in Europe volatile fruit flavors may be deliberately altered during distillation by a catalysis which favors ester formation. Thus the latent aroma of unripened fruit may be said to develop during recovery to a semblance at least of that found in the sun-ripened product.

I have talked a great deal about fruit juices for most of

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[4] "Studies on The Recovery of Essence from Florida Citrus Juices," by Donald A. Morgan and M. K. Veldhuis, R. K. Eskew and G. W. M. Phillips. Presented at the Twelfth Annual Meeting of the IFT at Grand Rapids, Michigan, June 9, 1952.

our experience has been in this field. To an ever increasing extent juices are being sold in the form of concentrates. If these concentrates are made by vacuum evaporation they will not yield juices of satisfactory quality unless the volatiles are restored. In the case of citrus juices this is of course done by adding some fresh cut-back juice to the concentrate. This has proven satisfactory because the chief flavoring constituent here is in the peel oil, contained in the cut-back juice. However, if fresh flavor is to be had in high-density (e.g. 7-fold) concentrates made from non-citrus juices, essence must be restored. If then the volatiles must be restored to the juice concentrates, how will these volatiles themselves ever appear on the market as essences for use by manufacturers of extracts, beverages, candy or other food products?

Volatile Fruit Concentrates from Preserve Manufacture

The answer may partly lie in our most recent work which covers the recovery of volatile fruit concentrates from the vapors given off from vacuum preserve kettles. We have shown such recovery to be feasible on an industrial scale. It would require replacing the jet condensers on the preserve kettles by surface condensers and the installation of tanks for storing condensates from different preserves. These condensates would then be processed in essence recovery equipment somewhat simpler in design than the arrangement we saw in Slide 2. Well made preserves from most fruits retain sufficient flavor and do not require restoration of the essence. Thus the larger preserve manufacturers may eventually have volatile fruit concentrates for sale. There is another source for apple essence at least, and that is the juice of the peels and cores of sound apples used in making vinegar. Volatile concentrate can be profitably recovered from such juice without impairing the quality of the vinegar and affording the manufacturer another source of income. Could not such concentrates also be recovered from the fresh pomace of other fruit processing operations? That is one of our proposed fields of research.

There are disadvantages to natural fruit flavors; they are generally more dilute and more difficult to standardize than imitation flavors. However, they remain unique in at least one regard—they derive from the fruit itself; and if properly derived can reproduce the flavor of that fruit. The Regional Laboratories were established to further the use of agricultural commodities, among them fruits. To this end we have attempted to make possible, improved fruit products through the recovery and use of their volatile components. I will welcome any suggestions you may have as to how the utility of this work can be extended by adapting it to the needs of your industry.

President Grant: We are indebted to you, Mr. Eskew, for that very fine presentation, and, as was announced earlier in the meeting, if anyone has any questions to ask, I am sure Mr. Eskew will attempt to answer them.

Is there anybody in the audience who has a question he wants to ask?

If not, if you will just give me your attention for a few minutes more, I would like to read these important announcements.

(President Grant read several announcements.)

President Grant: We will now adjourn.

(The meeting adjourned at twelve-ten o'clock.)

TUESDAY AFTERNOON SESSION

May 12, 1953

The meeting reconvened at two-ten o'clock in the Stratosphere Room of the Hotel Traymore, Atlantic City, New Jersey, President W. G. Grant presiding.

President Grant: Gentlemen, if you will please come to order we will start this afternoon's meeting.

Each year at our meetings, we are favored with representatives from some of the other trade associations, and this year is, of course, no exception.

We have with us this afternoon a man who has been with us many times before, and one whom we all enjoy seeing and hearing. I don't think that Mr. Schlotterer needs any introduction whatsoever. I don't believe there is anyone here who does not know him.

Without further word, I am going to ask Mr. Schlotterer, as Secretary of the Vanilla Bean Association of America, to come forward and speak to us at this time. (Applause)

Mr. Roy C. Schlotterer: Mr. Chairman and Members of the Flavor Extract Manufacturers' Association: On behalf of the Vanilla Bean Association, I wish to extend their best wishes for a very successful meeting.

It is a pleasure for me to again attend your convention. I am not chosen to give this report as the Secretary of the Association, but because I am sort of a neutral party. About two months before the Flavoring Extract Meeting, we put up an antenna and we tried to get a lot of different points of view.

This year, for some unknown reason, very few messages